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Indian Standard

METHOD OF TESTING CORROSION RESISTANCE OF METALLIC AND OTHER NON-ORGANIC COATINGS BY CORRODKOTE TEST

(First Revision)

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INDIAN STANDARDS INSTITUTION MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002,

Indian Standard

METHOD OF TESTING CORROSION RESISTANCE OF METALLIC AND OTHER NON-ORGANIC COATINGS BY CORRODKOTE TEST

(First Revision)

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Indian Standard

METHOD OF TESTING CORROSION RESISTANCE OF METALLIC AND OTHER NON-ORGANIC COATINGS BY CORRODKOTE TEST

(First Revision)

0. FOREWORD

- **0.1** This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 February 1985, after the draft finalized by the Metallic and Non-metallic Finishes Sectional Committee had been approved by the Structural and Metals Division Council.
- 0.2 This standard was first published in 1976. It has been felt necessary to revise this specification to bring it at par with international standardization. In this revision, the scope and field of application of this test has been extended to metallic and other non-organic coatings. However, the satisfactory corelation with service exposure should be thoroughly established before this test is made applicable to coating combinations other than copper + nickel + chromium and nickel + copper.
- **0.2.1** In this revision, specified tests, exposure duration and evaluation of corrosion resistance test stipulated in Indian Standard specifications have also been incorporated in Appendix A.
- **0.3** The corrodkote test was developed by the American Electroplators' Society. The results of this test corelate reasonably well for copper + nickel + chromium and nickel + chromium platings designed for relatively severe service conditions.
- 0.4 The requirements regarding corrosion resistance test for nickel + chromium and copper + nickel + chromium platings have been specified in various Indian Standards. Extensive experience has shown that the neutral salt-spray test demands longer periods and does not reproduce all the factors that may be involved under widely different conditions of exposure. The limitation of tests in natural environment and the need for quicker evaluation provide justification for the development of accelerated corrosion tests, such as the acetic acid salt

spray (ASS) the copper accelerated acetic acid salt spray (CASS) and the corrodkote (CORR) tests. Such tests have become necessary for inspection and quality control.

- 0.5 While preparing the revision of this standard, necessary assistance has been derived from ISO 4541-1978 (E) 'Metallic and other non-organic coatings corrodkote corrosion test (CORR Test)', published by the International Organization for Standardization.
- 0.6 In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors which influence the progress of corrosion, such as the formation of protective films vary greatly with the conditions encountered. The results obtained in the test should not, therefore, be regarded as a direct guide to the corrosion resistance of the tested material in all environments where these materials may be used. Furthermore performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.
- **0.7** In reporting the results of a tests or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS: 2-1960*.

1. SCOPE

- 1.1 This standard specifies the reagents, the apparatus and the procedure for assessment of the quality of metallic and related coatings by the Corrodkote procedure, in terms of evaluation of the corrosion resistance of such coatings.
- 1.1.1 This method is primarily applicable to coper + nickel + chromium and nickel + chromium electroplated parts.

2. PRINCIPLE

2.1 This test is carried out by applying a slury containing corrosive salts to the test specimens, allowing the slurry to dry, and exposing the specimens so coated with the slurry to a high relative humidity at a specified temperature for a specified period of time.

^{*}Rules for rounding off numerical values (revised). , .

3. PREPARATION OF REAGENTS AND CORRODKOTE SLURRY

3.1 Reagents for Slurry

- 3.1.1 Cupric Nitrate Solution Weigh exactly 2.50 g of cupric nitrate [Cu (No₃)₂ 3H₂O], and dilute with distilled water to exactly 500 ml in a volumetric flask.
- 3.1.2 Ferric Chloride Solution Weigh exactly 2.50 g of ferric chloride (FeCl₃·6H₂O), and dilute with distilled water to exactly 500 ml in a volumetric flask. Keep this solution in darkness in a flask fitted with a rubber or glass stopper, when not in use. The ferric chloride solution shall not be more than two weeks old, since older solutions become unstable.
- 3.1.3 Ammonium Chloride Solution Weigh exactly 50.0 g of ammonium chloride (NH₄ Cl), and dilute with distilled water to exactly 500 ml in a volumetric flask. This is the standard ammonium chloride solution reagent.

3.2 Preparation of Corrodkote Slurry

- 3.2.1 Measure exactly 7.0 ml of cupric nitrate solution and place it in a beaker (glass) of 250 ml.
- 3.2.2 Measure exactly 33.0 ml of ferric chloride solution and add it to the beaker containing cupric nitrate solution (see 3.2.1).
- 3.2.3 Measure exactly 10.0 ml of ammonium chloride solution and add it to the beaker containing cupric nitrate and ferric chloride solutions (see 3.2.2), total volume now in the beaker is 50 ml.
- 3.2.4 Weigh 30.0 g of water-washed ceramic grade kaolin and add it to the reagents in the beaker (see 3.2.3).
- 3.2.5 Mix the ingradients in the beaker thoroughly with a glass rod, stirring until a homogenous slurry is obtained and allow it to stand for about two minutes so that the kaolin becomes saturated. Mix the slurry by stirring with a glass rod just before using for application to the test specimens. (For stirring, use glass rod only as otherwise the slurry will attack any metallic stirrer.)
 - 3.2.6 Prepare a fresh batch of slurry for each batch of test specimen.

Note 1 — An alternate method of preparing the corrodkote slurry is as follows:

Dissolve 0.035 g of cupric nitrate [Cu (NO₃)₃·3H₂O], 0.165 g of ferric chloride (FeCl₃·6H₂O) and 1.0 g of ammonium chloride (NH₄Cl) in 50.0 ml of distilled

water and stirring into this solution 30.0 g of water-washed ceramic grade kaolin. Proceed further as in 3.2.5.

Note 2 — Reagents used as in 3,1.1 to 3,1.3 should be laboratory reagent grade.

4. APPARATUS

4.1 The apparatus shall consist of a non-condensing humidity chamber, specimen supports, provisions for heating the chamber and for air circulation in the chamber.

Note — For redevelopment of points of failure (see 10.3), a condensing type humidity chamber is used.

- 4.2 The design of the apparatus shall be such that the drops of moisture which might accumulate on the roof of the walls of the chamber or on the specimen supports, shall not fall on the specimens under test.
- 4.2.1 Suitable apparatus may be constructed from salt spray equipment as prescribed in Indian Standard, 'Recommended construction details of apparatus for spray cabinet useful for various salt spray tests (under preparation)', by eliminating the fog spray nozzles, substituting the test solution (salt solution) in the reservoir by (distilled) water, adding a manifold for bubbling air through the water in the reservoir, and adding a fan for circulating the air in the chamber with the fan-discharge directed across the surface of the water in the reservoir.

5. TEST SPECIMENS

- 5.1 Type of Specimens The type and number of test specimens to be tested as well as the criteria for evaluation of the test results, shall be as defined in the respective specifications covering the material or product being tested or shall be as mutually agreed upon by the purchaser and the supplier.
- 5.2 Preparation of Test Specimens—The test specimens shall be solvent cleaned prior to subjecting to this test, using a suitable solvent, such as ethanol, diethyl ether, acetone or petroleum ether. Solvents that are corrosive or that may deposit protective films shall not be used.
- 5.3 Application of Corrodkote Slurry The corrodkote slurry (freshly-prepared) shall be applied to the test specimens using a clean (paint) brush. Dip the brush in the corrodkote slury and, with a circular motion cover the test specimen completely. Then, smooth out the coating of slurry by brushing lightly in one direction. The wet film thickness shall not be less than 0.08 mm and not more than 0.2 mm. Allow the test specimens to dry at an ambient temperature and at a relative humidity lower than 50 percent for one hour before placing them in the non-condensing humidity chamber.

5.4 Protection of Cut Edges — If the test specimens are required to be cut from a larger coated article, the cutting should be carried out in such a way that the coating is not damaged, especially in the area adjacent to the cut. Care should also be taken to avoid contamination by swarf during cutting. Unless otherwise specified, the cut edges should be adequately protected by coating them with suitable medium that is stable under the conditions of the test, such as paint, varnish, wax or adhesive tape to prevent any galvanic effect between such edges and the adjacent coated or plated metal surfaces.

6. POSITION OF THE SPECIMENS DURING TEST

6.1 The position of the test specimens in the humidity chamber during the test is not critical provided that they do not touch each other or the walls, and the supports do not touch the test areas that have been coated with the corrodkote slurry.

7. CONDITIONS IN THE HUMIDITY CHAMBER

7.1 The exposure zone of the humidity chamber shall be maintained at $38 \pm 1^{\circ}$ C. The relative humidity of the exposure zone shall be maintained between 80 and 90 percent, so that condensation is not produced on any of the test specimens. Condensation on the roof and walls of the chamber is permitted provided it does not reduce the humidity in the chamber below the minimum level of 80 percent.

Note — A fan or blower may be used in the chamber to maintain uniform temperature and humidity. The amount of air-circulation in the chember necessary to maintain these conditions shall be determined for each chamber.

8. TEST CYCLE

- 8.1 Unless otherwise specified, for the coating or product being tested, the exposure in the humidity chamber shall be continuous for periods up to 16 hours. A single test period up to 16 hours shall be considered as one test-cycle. Centinuous operation implies that the humidity chamber be closed and operated continuously except for the short interruptions necessary to place, examine or remove test specimens. Operations shall be so scheduled that these interruptions are held to a minimum.
- 8.2 If more than one test cycle of 16 hours or less is specified, the test specimen shall be treated as specified in 10.1 and 10.2 after each test cycle. Fresh corrodkote slury shall be applied for each succeeding cycle. After the last test cycle, the test specimen shall be treated as specified in 10.1, 10.2 and 10.3.

9. PERIOD OF TEST

9.1 The period of each test cycle, as well as the number of test cycles required, shall be as specified in the relevant specification for the coating or the product being tested, or as mutually agreed to between the purchaser and the supplier.

10. TREATMENT OF TEST SPECIMENS AFTER TEST

- 10.1 Unless otherwise specified, for the material being tested, the test specimen shall be treated as described in 10.2. After the last test cycle, and if the test specimen is to be inspected between test cycles, the test specimen shall be treated, as described in 10.2 and 10.3.
- 10.2 Cleaning of Test Specimens Remove the test specimens from the humidity chamber. Examine the test specimens with the slurry intact. Clean the test specimens using fresh running water and a piece of clean cheese cloth or a suitable synthetic sponge to remove all the slurry. A mild abrasive (such as a water paste of tripoli or magnesium oxide) may be used to remove any tenacious adhering material (such as in case of zinc die castings).
- 10.3 Redevelopment of Points of Failure As the cleaning operations described in 10.2 remove any corrosion products, it is necessary to redevelop these points of failure. This may be done in several ways, such as exposure for 4 hours to the neutral salt spray test as specified in IS: 9844-1981*, exposure for 24 hours in a humidity chamber operated at 38°C and 100 percent relative humidity with condensation, or some other method which causes corrosion of the basis metal without any significant damage to the coating.

Note — Corrosion products visible on the test specimen with the corrodkote slury in place are not necessarily caused by points of failure in the coating. Particles of metal accidently deposited on the test specimen prior to exposure to humidity will also cause visible corrosion products. Care should be taken to avoid such contamination.

11. EVALUATION OF RESULTS

- 11.1 Criteria for the evaluation of the results of the test will usually be given in the specification for the coating or the product being tested. For most applications of the test, only the following need to be considered.
 - a) The appearance after the test;
 - b) The appearance after removing superficial corrosion products; and

^{*}Methods of test for corrosion resistance of electroplated and anodized aluminium coatings by neutral salt spray test.

c) Careful and immediate examinations for the extent of corrosion of the test specimen or for other failures (for example, pits, cracks, blistors, etc.) after redevelopment of points of failure as described in 10.3. The number and distribution of corrosion defects may conveniently be assessed by methods as specified in IS: 6009-1970*.

12. TEST REPORT

- 12.1 Unless otherwise specified in the specifications covering the coating or the product being tested, the test report shall contain the following information:
 - a) The specification of the coating or the product being tested;
 - b) The type of coating and the base with an indication of the surface finish of the coating;
 - c) The number of the test specimens of each coating or product subjected to the test;
 - d) The dimensions and shape of test specimens and the nature and area of the surface tested;
 - e) The preparation of the test specimens, including any cleaning treatment applied and any protection given to the edges or other special areas;
 - f) The method, if any, used to clean the test specimens after the test with, when appropriate, an indication of the loss of mass resulting from the cleaning operation;
 - g) The readings of the temperature maintained within the exposure zone of the humidity chamber;
 - h) The readings of the relative humidity maintained within the exposure zone of the humidity chamber;
 - j) The exposure period for each cycle and the number of test cycles employed for the test;
 - k) An indication of the precautions taken to verify that all the requirements stated in this Indian Standard have been adhered to:
 - m) The behaviour of any reference panels placed in the test chamber and exposed to the same conditions along with the test specimen; and
 - n) The results of all inspections.

^{*}Method for evaluation of results of accelerate corrosion tests.

APPENDIX A

(Clause 0.2.1)

SPECIFIED TESTS, EXPOSURE DURATION AND EVALUATION OF CORROSION RESISTANCE TESTS STIPULATED IN IS: SPECIFICATIONS

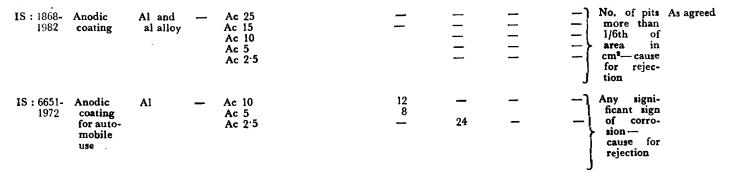
	SPECI-	COATING	Base	SERVICE	CLASSIFICATION No.	CORROSION TEST DURATION (HOURS)				EVALUATION	REMARKS	
	FIGATION			Grade No.	140.	CASS IS: 5528- 1985	ASS IS:6910- 1985	NSS IS: 9844- 1981	CORR IS: 8038- 1985			•
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	
	IS: 1068- 1985	Cu+Ni+Cr	Fe & steel	4	Fe/Cr20Ni30bCr mc/mp Fe/Cu20Ni30pCr r Fe/Cu20Ni25pCr mc/mp Fe/Cu20Ni30dCr r Fe/Cu20Ni25dCr mc/mp	24	144		2×16			
10		Ni+Cr	do	4	Fe/Ni40pCr r Fe/Ni30pCr mc/mp Fe/Ni40dCr r Fe/Ni30dCr mc/mp							
		Cu+Ni+Cr	do	3	Fe/Cu20Ni35bCr r Fe/Cu20Ni25bCr mc/mp Fe/Cu15Ni25pCr r Fe/Cu15Ni20pCr mc/mp Fe/Cu15Ni25dCr r Fe/Cu15Ni20dCr mc/mp	16	96	_	16 }	Minimum rating of 8 as per IS:6009 for acceptance		
		Ni+Cr	do	3	Fe/Ni40bCr r Fe/Ni30bCr mc/mp Fe/Ni30pCr r Fe/Ni25pCr mc/mp Fe/Ni30dCr r Fe/Ni25dCr mc/mp		•					

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		Cu+Ni+Cr Ni+Cr		2 2	Fe/Cu20Ni10bCr r Fe/Ni20bCr r	4	24		4) 	do		
		Cu+Ni+Cr Ni+Cr		1	Fe/Cu10Ni5bCr r Fe/Ni10bCr r	•	8	-	•	do	*Not suitable	
	IS: 4828- 1983	Cu+Ni+Cr	Zn and Zn-alloy	4	Zn/Cu20Ni35bCr mc/mp Zn/Cu20Ni35pCr r Zn/Cu20Ni25pCr mc/mp Zn/Cu20Ni35dCr r Zn/Cu20Ni25dCr mc/mp	24	144	_	2×18	Minimum rating of 8 as per IS: 6009-1970		
-				3	Zn/Cu20Ni35bCr r Zn/Cu20Ni25bCr mc/mp Zn/Cu20Ni25pCr r Zn/Cu20Ni20pCr mc/mp Zn/Cu20Ni25dCr r Zn/Cu20Ni20dCr mc/mp	16	96	-	16	for accep- tance		
				2 1	Zn/CuNi15bCr r Zn/CuNi8bCr r	4	24 8		4		Indercoat of Cu or brass, Min 8 µm	
	IS: 4942- 1983	Ni + Cr	Al and Al alloys		Al/Ni50dCr r Al/Ni35dCr mc/mp	24	144	_	2×16	Minimum rating of 8 as per IS: 6009-1970 for accep- tance	Cu under- coat desir- able for some alloys; but with no reduction of Ni-layer	IS : 8
				3	Al/Ni35pCr r Al/Ni30pCr mc/mp Al/Ni30dCr r Al/Ni25dCr mc/mp	16	96	_	16	do	Continued)	8038 - 1985

agreed

12



b — bright nickel;
p — dull or semi-bright nickel;
d — double or triple layer nickel;
r — regular chromium 0·3 mm, Min;
mc — micro-crack chromium 0·3 m, Min; and
mp — micro-porous chromium 0 3 m, Min.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	Unit	Sумвоц	
Length	metre	m	
Mass	kilogram	kg	
Time	second	\$	
Electric current	ampere	A	
Thermodynamic temperature	kelvin	K	
Luminous intensity	candela	cd	
Amount of substance	mole	mol	
Supplementary Units			
QUANTITY	Unit	Symbol	•
Plane angle	radian	rad	
Solid angle	steradian	sr	
Derived Units			
QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$i N = 1 \text{ kg.m/s}^3$
Energy	joule	J	1J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	webe r	Wb	1 Wb = 1 V.s
Flux density	tesla	T	$1 T = 1 Wb/m^2$
Frequency	hertz	Hz	1 Hz = 1 c/s (s-1)
Electric conductance	siem ens	S	1 S = 1 A/V
Electromotive force	volt	v	1 V = 1 W/A
Pressure, stress,	pascal	Pa	$1 Pa = 1 N/m^2$